NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
WASHINGTON, D.C. 20546

REPLY TO
ATTN OF: GP

March 30, 1971

TO: USI/Scientific & Technical Information Division
Attention: Miss Winnie M. Morgan

FROM: GP/Office of Assistant General
Counsel for Patent Matters

SUBJECT: Announcement of NASA-Owned
U.S. Patents in STAR

In accordance with the procedures contained in the Code GP to Code USI memorandum on this subject, dated June 8, 1970, the attached NASA-owned U.S. patent is being forwarded for abstracting and announcement in NASA STAR.

The following information is provided:

U.S. Patent No. : 3,396,303

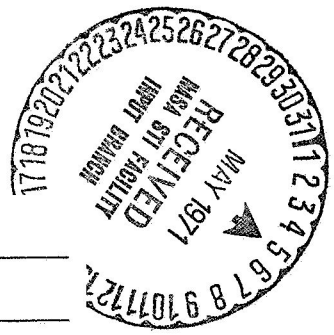
Corporate Source : Lewis Research Center

Supplementary
Corporate Source :

NASA Patent Case No.: XLE-04788

Gayle Parker

Enclosure:
Copy of Patent



N71-22987
(ACCESSION NUMBER)

4
(PAGES)

(NASA CR OR TMX OR AD NUMBER)

08
(CODE)

09
(CATEGORY)

FACILITY FORM 602

NASA-HQ

N71-22987

Aug. 6, 1968 W. A. GORDON 3,396,303

ARC ELECTRODE OF GRAPHITE WITH BALL TIP

Filed March 21, 1966

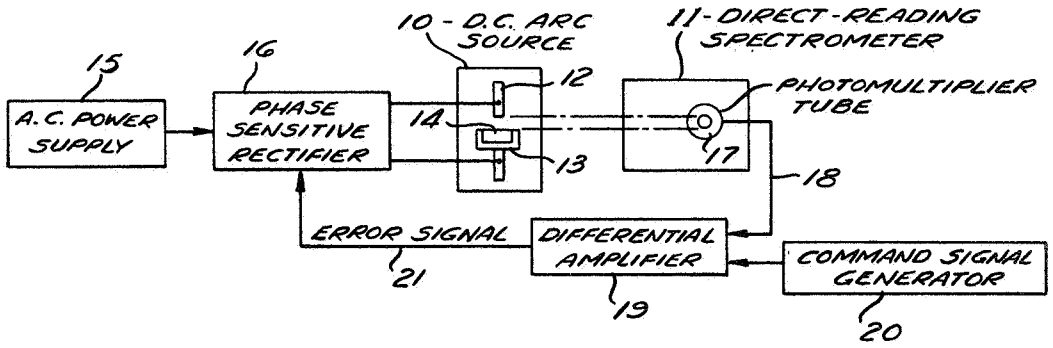


Fig. 1

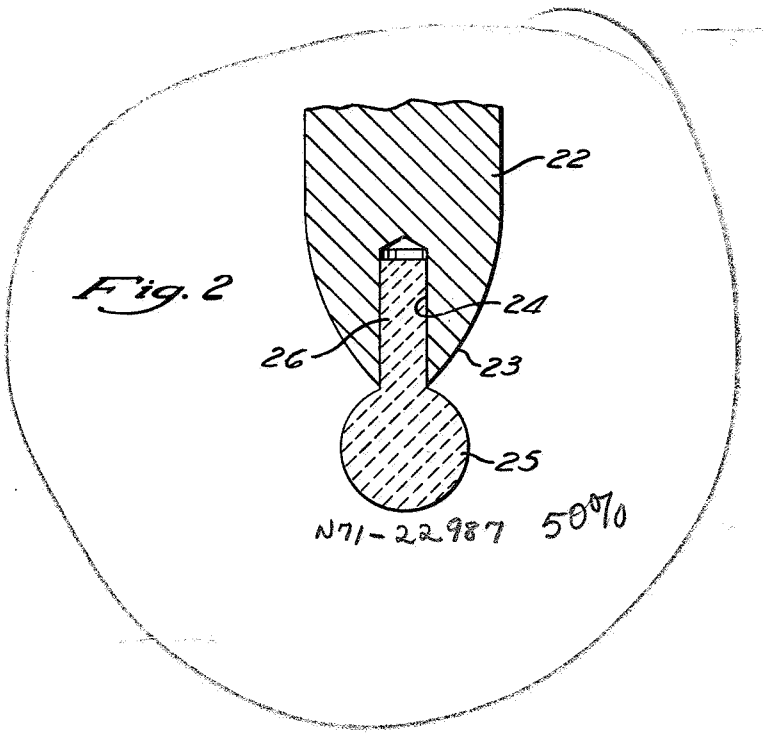


Fig. 2

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United States Patent Office

3,396,303

Patented Aug. 6, 1968

3,396,303

ARC ELECTRODE OF GRAPHITE WITH BALL TIP

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Filed Mar. 21, 1966, Ser. No. 537,617

3 Claims. (Cl. 313—352)

ABSTRACT OF THE DISCLOSURE

An arc source for emission spectroscopic analyses in which the cathode electrode comprises a graphite post having a globular tantalum tip.

The invention described herein was made by an employee of the United States Government and may be manufactured and used by or for the Government for governmental purposes without the payment of any royalties thereon or therefor.

This invention relates generally to apparatus for controlling an energy source for spectral analysis and more particularly to such apparatus for controlling a D.C. arc source for an emission spectrograph or spectrometer.

In making trace analyses by emission spectroscopy, a major source of difficulty has been the arc source used to excite the sample material. Commonly, the sample is held in a graphite crucible to provide the anode of the arc source, and a suitable potential is applied between this anode and the cathode of the arc source to produce an arc discharge for vaporizing the sample and for exciting the atomic spectra of the sample to produce light from which the trace analysis may be made by known techniques.

Ideally, the arc source in such equipment should have both high sensitivity and high precision. It has been recognized that a D.C. arc source is extremely sensitive, but prior to the present invention there has been no entirely satisfactory way to achieve the desired precise control over the output energy of the D.C. arc source. In practice this has meant that the analytical spectroscopist must have available to him a variety of different arc sources, including a D.C. arc source, a spark source, various combinations of the two, and high voltage and low voltage A.C. arc sources. In the use of such arc sources the analytical spectroscopist must, in any specific instance, sacrifice precision for sensitivity, or vice versa.

The present invention is directed to an apparatus which avoids these difficulties and disadvantages by providing an improved arrangement for precisely controlling the output energy of an energy source for spectral analysis. As applied to emission spectroscopy, the present control regulates the output energy of a D.C. arc source, so that the user may take advantage of the high sensitivity inherent in the D.C. arc source, without, however, sacrificing the desired precision.

In accordance with the preferred embodiment of this invention, these objectives are achieved by controlling the vaporization rate of the sample, and therefore the output energy of the D.C. arc source, in accordance with a programmed signal, either a constant or a time-dependent command signal, thereby making the output energy of the D.C. arc source independent of transient events which may take place in the arc source. The output energy of the D.C. arc source is compared with a programmed command signal and the difference between

them produces an error signal which regulates the direct current supplied to the arc source, so as to regulate the latter's output energy in accordance with the programmed command signal.

Another aspect of the present invention is directed to stabilizing the arc of the D.C. arc source so that it is a steady, rather than a flickering, source of light. This is achieved by providing a novel and improved cathode structure for the arc source having a refractory metal tip which enables a more stable arc discharge to be maintained.

While the present invention is particularly applicable to the analysis of trace constituents in a sample by emission spectroscopy, it is also advantageous for use in determining major constituents of the sample, such as alloy constituents, by emission spectroscopy. Also, it may be used in mass spectrometry and in atomic absorption spectroscopy, if desired.

It is a principal object of this invention to provide a novel and improved apparatus for controlling the output energy of an energy source for spectral analysis.

Another object of this invention is to provide such an apparatus which controls the output energy of the energy source in accordance with a constant or time-programmed command signal, thereby eliminating the effects of transient events in the energy source.

Another object of this invention is to provide such an apparatus which is particularly useful for controlling the output energy of a D.C. arc source for a spectrograph or spectrometer.

Another object of this invention is to provide such an apparatus having a novel and improved cathode construction for stabilizing the arc in a D.C. arc source for a spectrograph or spectrometer.

Further objects and advantages of this invention will be apparent from the following detailed description of a presently-preferred embodiment thereof which is shown in the accompanying drawing.

In the drawing:

FIGURE 1 is a schematic circuit diagram of a presently-preferred embodiment of the present apparatus; and

FIGURE 2 is an enlarged fragmentary longitudinal section of the cathode in the arc source in the FIG. 1 apparatus, in accordance with the preferred embodiment of this invention.

Referring first to FIG. 1, an energy source in the form of a D.C. arc source **10** is associated with a direct reading spectrometer **11** of known construction, the details of which will be understood by those skilled in the art and therefore need not be described in detail.

The D.C. arc source **10** includes a cathode **12** and an anode constituted by a graphite holder **13** and the sample **14** in the holder. The cathode and anode are in spaced confronting relationship with respect to one another. A D.C. source for producing the arc across these electrodes is provided by an A.C. power supply source **15** and a phase-sensitive rectifier **16** connected between the output of this A.C. power supply source and the electrodes.

The spectrometer **11** is provided with a photomultiplier tube **17** of known construction which senses the light output of a selected spectral line which is emitted by the arc produced by the source **10**. That is, the light energy produced by atomic emission of a selected element in the arc is the input signal to this photomultiplier tube **17**. The output signal from the photomultiplier tube **17** is an electrical signal on line **18** having a magnitude substantially proportional to the light energy produced by the arc. This output signal is applied to one input terminal of a differential amplifier **19** of known design.

In accordance with the present invention a second input signal to this differential amplifier is generated by a command signal generator **20** which may be either a time-programmed function generator or a constant signal generator of known design, which produces either a time-dependent or constant command signal in accordance with a predetermined program. The time-dependent function generator may be a curve following programmer of any suitable design, or it may be a function potentiometer of the general type shown at page 253 of the book, "Electronic Analog Computers" by Korn et al., published by McGraw-Hill Book Company, New York, 1952, for example.

In the differential amplifier **19** the output signal from the photomultiplier tube **17** is compared with the output signal from the command signal generator **20**. The difference between the output signal from the photomultiplier tube **17** and the signal from the command signal generator **20** produces an error signal on the output line **21** from the differential amplifier **19**. The sign and the magnitude of this error signal depend upon the respective magnitudes of the command signal and the output signal from the photomultiplier tube. This error signal is applied to the phase-sensitive rectifier **16** to control the magnitude of the latter's output, which provides the current for the arc source **10** and therefore controls the rate at which the sample **14** is vaporized.

The arrangement is such that if the output signal from the photomultiplier tube **17** has a higher amplitude than that of the signal from the command signal generator **20** (indicating that the output energy of the arc source **10**, which is determined by the rate at which the sample **14** is being vaporized into the arc, is higher than that called for by the command signal), then the error signal applied to the phase-sensitive rectifier **16** will reduce the latter's output, thereby reducing the direct current applied to the arc source **10** so as to reduce the latter's output energy to the value called for by the command signal. Conversely, if the output signal from the photomultiplier tube **17** has a smaller amplitude than that of the signal from the command signal generator **20** (indicating that the output energy of the arc source is lower than what is called for by the command signal), then the error signal applied to the phase-sensitive rectifier **16** will increase the latter's output, thereby increasing the direct current applied to the arc source **10** so as to increase the latter's output energy to the value called for by the command signal.

With this arrangement, therefore, the output energy of the arc source **10** is regulated in accordance with the signal of the command signal generator **20**. This gives extremely precise control over the output energy of the arc source while at the same time retaining the advantages of the high sensitivity which is inherent in the D.C. arc source, as compared with other types of arc sources.

From the foregoing it will be evident that the present control arrangement achieves the desired objective of controlling the vaporization rate of the sample in a manner which enables the reproduction of that same programmed vaporization rate for subsequent samples. This is in contrast to previous proposals to provide a constant input current to the arc, which failed to achieve the desired objective because the vaporization of the sample can itself change the arc current. Similarly, prior proposals to hold the arc current constant regardless of the rate at which vapors enter into the arc fail to achieve the objective of providing a reproducible vaporization rate of the sample.

While the preferred embodiment is aimed at controlling the vaporization rate of the analytical specimen, it may be desirous to control the excitation of elements subsequent to their entering the arc column. This may be accomplished by imposing control on the effective excitation temperature of the arc column. A further embodiment, therefore, of the control arrangement is where the reference radiation from the arc in the arc source **10** is comprised of two spectral lines, the ratio of which is a function of the effective excitation temperature of the arc. The electrical ratio of the two lines is the input signal on line **18** to the differential amplifier **19**, and this signal is compared to the signal from the command signal generator **20**. The matching of the ratioed reference signal to the command signal may then be accomplished by appropriate adjustments in arc current, gas pressure, gas composition or other parameters.

While the present control arrangement has been described with reference to the regulation of a D.C. arc source for an emission spectrograph or spectrometer, it may also be used to control the variable energy source in atomic absorption spectroscopy. For example, if the sample being analyzed is in the form of a solution being aspirated into a flame which provides the excitation of the sample, the present control may be used to regulate the aspiration pressure or in some other way to control the rate of aspiration of the sample in accordance with a programmed control signal.

Another aspect of the present invention is directed to stabilizing the arc in the source **10**. For trace analysis of a sample in a static inert atmosphere, such as argon, the free running D.C. arc has been found to be unstable, compared to its performance in air. In argon the arc produces a noticeable anode-flame, composed of plasma streaming toward the anode. This anode-flame tends to revolve erratically about the axis of the electrode because of convection and buoyancy effects arising from the thermal gradients between the arc column and the surrounding atmosphere. This instability of the arc column leads to poor precision of measurement.

Referring to FIG. 2, in accordance with the present invention this difficulty is overcome by the provision of a novel cathode in the arc source **10** of FIG. 1. This cathode includes a graphite rod or post **22** having a tapered end **23** in which an axial recess **24** is drilled. In the arc source **10** (FIG. 1) this tapered end of the post is disposed toward the anode. The cathode also has a refractory metal tip, preferably of tantalum, which presents a generally cylindrical globular bead **25** extending across this end of the graphite rod and an integral stem **26** which is received tightly in the recess **24** to secure the refractory metal tip to the graphite post. In one practical embodiment the refractory metal bead **25** is about 2 mm. in diameter.

When used in combination with a conventional anode arrangement in the D.C. arc source, it has been found that the cathode spot, when the arc is maintained, is spread over substantially the entire surface of the bead **25**. Consequently, the current density and the magnetic fields are reduced and the plasma streaming, or anode-flame, is greatly diminished or completely eliminated, thus providing a highly stabilized arc column.

When used in emission spectrographic trace analysis, this novel cathode improves the reproducibility of line and background intensities and substantially eliminates arc wandering or fluctuations as a source of error without reducing the sensitivity of measurement for either solid or liquid samples. The cathode, constructed as described, may be used for hundreds of trace analyses without the need for special treatment between exposures.

The arc is initiated preferably by using a spark igniter to ignite the graphite post **22**. After about two seconds the refractory metal bead **25** will be sufficiently hot to thermally emit, at which time it takes over as the cathode.

While a presently-preferred embodiment of this invention has been described in detail with reference to the accompanying drawings, it is to be understood that various modifications, omissions and refinements which depart from the particular embodiment disclosed may be adopted without departing from the spirit and scope of the present invention.

I claim:

1. In a D.C. arc source for a spectral-measuring instrument having an anode and a cathode in spaced, confronting relationship to each other, the improvement which

comprises a cathode structure comprising a graphite post and a refractory metal tip secured to said post and extending across the latter's end which is disposed toward the anode, said post being of progressively reduced cross section towards said end, and said refractory metal tip presenting a globular bead extending across said end of the post.

2. The cathode of claim 1 wherein an integral stem is formed on said globular bead, said post having a recess in said end, said stem being disposed in said recess whereby said globular bead is retained on said post.

3. The cathode structure of claim 1, wherein said refractory metal tip is of tantalum.

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